LABORATORY EVALUATION OF AN IN-SITU COATING PROCESS FOR MITIGATION OF LEAD AND COPPER IN DRINKING WATER

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ABSTRACT

Water pipe failures (leaks) within buildings are an ongoing problem at many large installations such as military bases, universities, and industrial complexes. Such failures can cause severe, costly damage to building interiors, personal property, and electronic equipment. Most failures in metallic potable water pipes may be induced by certain constituents in the water and/or poor workmanship practices. Water temperatures, pressures, and velocities are also factors in the corrosion process. In order to mitigate water-side corrosion and comply with the 1986 Safe Drinking Water Act and the National Primary Drinking Water Regulations for lead and copper, 40 CFR 141.82, an alternative to chemical treatment was developed. The preliminary results of in-situ pipe coatings for small diameter building plumbing are presented.

INTRODUCTION

Army installations must comply with the increasingly stringent drinking water quality standards enacted at the Federal level and enforced by State regulations. The Safe Drinking Water Act (SDWA) of 1974 required the U.S. Environmental Protection Agency (USEPA) to develop a list of maximum contaminant limits (MCLs) for inclusion in the National Primary Drinking Water Regulations (NPDWR). A September 1986 amendment to the SDWA banned the use of lead in public water system pipes, solder, and flux. On 7 June 1991, the USEPA finalized these regulations with a requirement of an MCL for lead concentration of 0.015 mg/L (2.00^10-6 oz/gal) measured in the ninetieth percentile taken from cold water kitchen faucets following a 6 to 8-hour stagnation time. In September 1992, the USEPA finished Volume II of the Lead and Copper Rule (LCR), which is the guidance manual on corrosion control treatment.

Much attention has focused on the costly remediations required when the lead action level is exceeded. This issue plays a significant role in the national debate over unfunded environmental mandates, and more specifically, in the search for cost-effective ways to ensure that drinking water at Army installations meets all standards for quality and compliance with applicable laws. Two possible strategies to ensure that drinking water meets current standards are by chemical treatment and by application of coatings or linings to pipes or tubes to mitigate corrosion or plumbosolvency (the leaching of lead into water). In addition to plumbosolvency, copper tubes and fittings fail by pitting corrosion, concentration cell corrosion and galvanic corrosion as a result of both water chemistry and workmanship. Chemical treatment alone will not mitigate corrosion induced failure where poor workmanship has been identified as the primary cause of failure. The use of an in-situ coating or lining can be utilized as an alternative to chemical treatment to mitigate these types of corrosion induced failures. An example of corrosion induced water-side pipe failure within building plumbing systems that could be mitigated by in-situ coating is shown in Figure 1. This study was undertaken to evaluate the effectiveness of an in-situ epoxy coating in comparison with a proven chemical treatment for potable water.

The purposes of this study were to initiate operation of the Water Treatment Test Facility (WTTF), and to execute a 12-week test program. This study evaluated the effectiveness of an insitu epoxy coating for the mitigation of lead and copper corrosion in comparison to both a control and a proven, effective chemical treatment (zinc orthophosphate) for potable water.

EXPERIMENTAL PROCEDURE

The initial phase of the laboratory investigation was to determine whether a coating or lining could be applied in-situ to existing small diameter (1"-2") pipes or tubes. The experimental procedures consisted of fabricating copper pipe specimens, abrasively cleaning the interior surface followed by either blowing through or depositing the 100% solids epoxy coating. The coating application loop is represented in Figure 2. The epoxy coating was then air cured. The evaluation consisted of sectioning the specimens and measuring the coating thickness to determine uniformity of each application technique, in addition to taking weekly water analysis samples to determine lead ion contents.

The water entering the water treatment test facility can be altered mechanically and chemically to produce a water with the desired concentrations of hardness, alkalinity, pH, calcium, etc. Several different water qualities (up to four) can be evaluated

during each daily cycle by using a computer to control sequencing of valves, pumps, etc. Provisions have been made for testing up to four different chemical treatments for each of the water qualities. Figures 3, 4, and 5 provide detailed schematics of various elements of the WTTF.

This study employed only two water qualities. One was softened water (<2.0 mg/L hardness as CaCO₃) with an alkalinity of ~200 mg/L and a flowing pH of approximately 7.0, and the second was a hard water (municipal supply, $\sim 80 \text{ mg/L}$ as $CaCO_3$) with an alkalinity of 200 mg/L and a flowing pH of 7.5. The original intent was to operate the hard water system with a pH of 8.0, but the water pH was not stable in that range. The incoming municipal water has a nominal pH of 8.8 to 9.0, so the pH is first lowered and the alkalinity neutralized by the addition of sulfuric acid. Soft water test loops are first passed through a water softener to remove most of the hardness. After the acid addition, solutions of first sodium bicarbonate and then sodium hydroxide are injected into the test loop to raise the alkalinity and pH to the desired levels. There were three water treatment schemes, a control with no coating, in-situ epoxy coating, and zinc orthophosphate. The two flow rates employed were 5 and 3 feet per second (fps). Each of the water qualities (WQ) were tested at two velocities (3 and 5 fps), the maximum for copper pipe in a typical building flow is 4 fps. This resulted in four designated water qualities:

- 1. WQ 1, hard water, 5 FPS
- 2. WQ 2, soft water, 5 FPS
- 3. WQ 3, hard water, 3 FPS
- 4. WQ 4, soft water, 3 FPS

Water flowed through each of the 12 legs for 2 hours each day. Each of the twelve legs in operation had a 3½-in. long copper specimen with ½-in. coating of 50/50 tin-lead solder with a thickness of .03"±.005" and 3 in. long copper specimens. Since the loops are constructed of PVC pipe, these pipe specimens are the only potential source of lead and copper in the system except the incoming city water.

Samples were taken weekly following an 8-hour standing time from each of the twelve legs and analyzed for lead, copper, total organic carbon (TOC), orthophosphate, zinc, methyl orange alkalinity (M-alkalinity), hardness, pH, and temperature. Source water for the loops was Champaign-Urbana tap water (Northern Illinois Water Corporation). This was monitored weekly for copper, TOC, orthophosphate, and zinc. The soft and hard water supplied to the legs was monitored for background concentrations in all of the species listed above. Oxygen and temperature levels during operation were recorded by on-line instruments.

The uncoated copper specimens were removed for corrosion weight loss measurements.

DISCUSSION OF RESULTS

ANALYTICAL DATA

One concern with the use of the in-situ epoxy coating was the possible decomposition of the coating, which might result in the release of organic compounds. The supply water was monitored for background TOC levels, and the average value for the 12-week period was 1.8 mg/L. The average for the four legs that used the in-situ epoxy coated specimens was 1.7 to 1.9 mg/L, indicating no significant decomposition of the epoxy coating. Neither the flow rate (5 fps and 3 fps), nor the water quality had any apparent impact on the lead or copper concentrations. The temperature was relatively constant throughout the course of the experiment, with a range of 16.9 to 20.3 °C, and an average value of 17.9 to 18.6 °C in the individual legs. The pH for the standing samples in all of the legs was constant at approximately 7.0 (±0.3).

Lead corrosion was obviously controlled by water treatment by using a zinc orthophosphate inhibitor, since very few of the samples contained lead concentrations above the detection limit. Copper concentrations were measurable for all 12 weeks in each of the four legs using zinc orthophosphate. The average concentration of copper was highest (0.51 mg/L) in the soft water, 3 fps leg. The average concentration of copper in the other three legs was very consistent at 0.41, 0.43, and 0.44 mg/L. None of the copper concentrations exceeded the USEPA 1.3 mq/L MCL. The average TOC concentration in these four legs was very similar to the coated specimen legs, ranging from 1.7 to 2.0 Zinc concentrations averaged between 0.86 and 0.96 mg/L, and the average orthophosphate concentrations ranged from 1.52 to 1.91 mg/L.The zinc orthophosphate treatment provided satisfactory corrosion inhibition of lead and copper.

Corrosion rates for both lead and copper was highest in the control legs. The soft water, 5 fps leg, had the lowest lead levels, averaging 1.04 μ g/L. The other three legs were more consistent with each other, averaging 2.10 to 2.74 μ g/L. Hard water showed higher Pb corrosion rates than soft water for lead at both flow rates. However, the average copper concentrations

were more consistent, ranging from 1.03 to 1.26 mg/L for the four control legs. Copper concentrations exceeded the MCL of 1.3 mg/L in 41 percent of the samples from these legs.

Once again, TOC concentrations were comparable in both the insitu epoxy coated specimen legs and the zinc orthophosphate treated legs, ranging from 1.8 to 2.0 mg/L. Champaign-Urbana tap water was used as the supply for these loops, and was monitored for copper, TOC, orthophosphate, and zinc. Zinc and copper concentrations were below instrument detection limits for all of the samples analyzed. The TOC concentrations averaged 1.8 mg/L for the duration of the test run. Trace amounts of orthophosphate recorded in two samples, may have been due to system pH upsets that resulted in a release of phosphate from existing deposits on the distribution piping (a dissolution of water chemistry originated deposits).

Flowing water samples from the hard and soft water supply loops were analyzed during most of the 12-week period for the same analyses as the legs. Oxygen concentrations averaged 3.6 mg/L in the soft water loop and 3.8 mg/L in the hard water loop. The temperatures were, as expected, much lower in the flowing samples, averaging near 10 $^{\circ}\text{C}$ for both loops. The copper, zinc, and lead concentrations in the loop were found to be at or below the detection limit.

WEIGHT LOSS DATA

Corrosion weight loss measurements were conducted on the 3-in. copper specimens installed in the zinc orthophosphate and control The corrosion rate was somewhat higher for the soft water than the hard water for both velocities in the control and treated legs. The 5 fps velocity legs had higher corrosion rates than the comparable 3 fps legs for three of the four water quality/velocity combinations. The soft water legs treated with zinc orthophosphate had the same corrosion rates for both the 3 and 5 fps legs. The effect of velocity on the corrosion rate of copper was obvious in the control legs. The copper corrosion rate in MPY (millimeters penetration/year) for the 5 fps legs in both the hard and soft waters was almost 20 percent higher than in the 3 fps legs. The recommended maximum velocity for copper tube in potable water systems is 4 fps; flow rates higher than that can cause an increase in corrosion rates. The copper corrosion rates for the four legs using zinc orthophosphate ranged from 0.34 to 0.49 MPY, and the corrosion rates in the control legs were 0.95 to 1.19 MPY. Copper corrosion rates of 1.0 MPY are much higher than desired for potable water systems, the desired corrosion rate is less than 0.5 MPY. Corrosion weight loss determinations were not performed on the 3½ in. specimens since ½ in. of the inside is coated with 50/50 tin-lead solder,

and it would be difficult to determine how much weight loss was attributable to copper and how much was lead or tin.

WILCOXON SIGNED RANK TEST

The Wilcoxon Signed Rank Test (Wilcoxon) is a nonparametric statistical analysis comparing two related (dependent) samples. The Wilcoxon takes into account the size of the rank order differences within pairs of data, as opposed to the numerical values of the differences. Paired data were examined among three different water treatment conditions (epoxy coating, zinc orthophosphate, and control) under four different water quality conditions. Table 1 summarizes the water treatments and qualities. The Wilcoxon was applied to look for differences among the three different water treatments.

In this experiment, copper, lead, and total organic carbon (TOC) values in water were recorded. The Wilcoxon was performed separately for each of these three elements. For the purpose of reading the tables, water quality is denoted by WQ, and water treatment is denoted by WT. The numbers corresponding to the water treatments and qualities are designated in Tables 1 and 2. The Wilcoxon tables are broken down into three sections:

- 1. Counts of Differences: This section presents the number of times element values from a given WQ and WT (listed along the left-hand column) are greater than the values for one of the other WTs (listed along the top row).
- 2. Z: This section presents the sum of the signed ranks divided by the square root of the sum of the squared ranks. This statistic is given meaning by the probability value obtained in statistical tables.⁴
- 3. Two-Sided Probabilities: The statistical significance to the corresponding Z-value is given in this section. A probability of 1.000 means the paired rankings are indistinguishable from one another and the differences between them are insignificant. A probability of 0.001 means there is a 99.9 percent probability the paired rankings are distinguishable and significantly different.

Table 2 summarizes the Wilcoxon for copper, lead, and TOC values.

Table 3 summarizes the results shown in Table 2, and ranks the water treatments for the reduction of each element. The data in Table 3 shows that the epoxy coating was the most effective for reducing copper concentrations in the pipe loop. Both the epoxy coating and the zinc orthophosphate were effective in reducing lead values, and both seemed to work equally well in comparison

with the control. However, all three water treatments were statistically indistinguishable from one another in reducing TOC values. None of the water treatments stood out as a good agent for the reduction of total organic carbon in water.

VISUAL OBSERVATIONS

Visual observations were made for all of the 3½-in. and 3-in. specimens after completing the test run, and before making any weight loss determinations. The exterior surface of each specimen was discolored, indicating there had been some seepage of water between the specimen and the holder. The specimens designated by C are the 3-in. copper specimens and those designated by P are the 3½-in. copper and tin-lead solder specimens. Table 4 lists the visual observations.

CONCLUSIONS

The results of this study clearly indicate that the in-situ epoxy coating provides an effective alternative to conventional chemical treatment for the prevention of lead and copper metal release in a system modeled to simulate a home plumbing system. Lead concentrations were lower than the USEPA MCL for all of the samples, but this was probably due to the very small surface area of lead available. The control samples had measurable lead concentrations in most samples (>80 percent), with three of the legs averaging more than 2 ug/L lead for the standing samples. The zinc orthophosphate and in-situ epoxy coating legs all had only occasional (<20 percent) lead concentrations above the detection limits. Copper concentrations were very high in the control legs, having average copper concentrations near the USEPA MCL of 1.3 mg/L (1.03-1.26 mg/L) for each leg, with ~41 percent of the samples exceeding the MCL. The zinc orthophosphate exhibited a significant improvement in the copper concentrations found in the standing samples for all water qualities, with none of the samples exceeding the MCL. The average copper concentrations varied from 0.41 to 0.51 mg/L. The in-situ epoxycoated legs showed an even more dramatic reduction of copper levels than the zinc orthophosphate treatment, with only one of the 48 samples having a copper concentration (0.030 mg/L) above the detection limit of 0.006 mg/L.

This study also initiated operation of the WTTF, and determined its viability as a test facility to simulate a variety of water qualities in a home plumbing system. The WTTF operated reliably over the course of the 12-week study, which gathered valuable information on operating procedures. Comprehensive information on the operation of the loop, computer programs, and equipment

specifications can be found in the Army Water Treatment Test Facility Operation Manual.

Table 2. Summaries of the Wilcoxin for copper, lead, and TOC values.

Water	aries of the Wilcoxin for cop Copper	TOC	
	Соррег	Lead	
Quality WQ1: "hard" water, 5 fps.	WT1 was the best treatment for this WQ, having a 99.8 percent significance over WT2 and WT3. WT2 ranked second, also having a 99.8 percent significance over WT3.	The results for comparing WT1 and WT2 were not distinguishable. However, both WT1 and WT2 were significantly better than WT3 (over 99 percent).	There was no significant difference between WT1 and WT2, as well as between WT2 and WT3. The results for WT1 and WT3 were 96.5 percent distinguishable, with WT1 prevailing.
WQ2: "soft" water, 5 fps.	The results for WQ2 were almost identical to WQ1. WT1 had over a 99 percent significance over WT2 and WT3. WT2 had a 99.7 percent significance over WT3.	The results were similar to the WQ1 results. WT1 and WT2 were not distinguishable, but both WTs prevailed over WT3.	None of the three water treatments were statistically distinguishable from one another for this water quality.
WQ3: "hard" water, 3 fps.	The results were exactly identical to the results obtained for WQ1. WT1 was the best, followed by WT2 and WT3.	In this case, WT1 and WT2 were distinguishable, with WT2 having a 95.7 percent significance over WT1. Both WT1 and WT2 were significantly better than WT3 (over 95 percent).	The results for WQ3 were the same as for WQ2. None of the three water treatments were statistically distinguishable from one another.
WQ4: "soft" water, 3 fps.	Once again the results were similar to WQ1. Order of performance: WT1, WT2, WT3.	WT1 had a slight significant edge over WT2 (92 percent), and WT3 had the most number of larger lead values, placing it last among the three water treatments.	For WQ4, WT2 was slightly significantly different over WT1 (91.9 percent). The remaining results were not distinguishable.

Table 3. Wilcoxon signed rank test results.

Water Quality	Copper Water Tre	eatment Rank	Lead Water Trea	tment]		TOC Water Treatment Rank
WQ1	WT1 WT2	2 WT3	WT1, WT2	WT3		WT1, WT2, WT3 — —
WQ2	WT1 WT2	2 WT3	WT1, WT2	WT3		WT1, WT2, WT3 — —
WQ3	WT1 WT2	2 WT3	WT2	WT1	WT3	WT1, WT2, WT3 — —
WQ4	WT1 WT2	2 WT3	WT1	WT2	WT3	WT1, WT2, WT3 — —

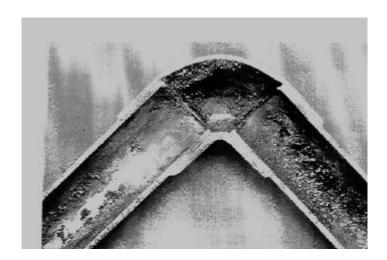


Figure 1. Copper tube showing workmanship defect, i.e., unreamed tube ends, solder globs, and excess solder flux.

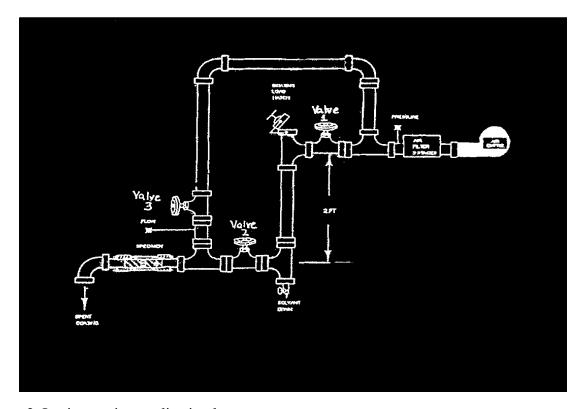


Figure 2. In-situ coating application loop.

Table 1. Sample Identifications

Sample	Water	Water	Treatment
Identification	Quality	Treatment	
A-1	1	1	Coating
I-1		2	Zinc Orthophosphate
R-1		3	Control
D-3	2	1	Coating
L-2		2	Zinc Orthophosphate
S-1		3	Control
F-1	3	1	Coating
M-1		2	Zinc Orthophosphate
V-1		3	Control
G-1	4	1	Coating
P-2		2	Zinc Orthophosphate
W-1		3	Control

¹ "Minimization of Lead Corrosion in Drinking Water," *Materials Performance* (August 1990), pp 45-49.

² U.S. Environmental Protection Agency (USEPA), *Lead and Copper Rule Guidance Manual Volume II: Corrosion Control Treatment*, EPA 811-B-92-002 (USEPA, September 1992).

 $^{^{3}}$ 1 ft = 0.305 m; 1 in. = 25.4 m.

⁴ Vincent F. Hock, Henry Cardenas, Kent W. Smothers, and Eric D. Zelsdorf, *Control of Plumbosolvency in Building Plumbing Supplies*, Technical Report (TR) 96/74/ADA315200 (U.S. Army Construction Engineering Research Laboratories [USACERL], July 1996).